

50X1-HUM

~~CONFIDENTIAL~~ ~~RESTRICTED~~
 CLASSIFICATION ~~RESTRICTED~~ ~~RESTRICTED~~
 SECURITY INFORMATION
 CENTRAL INTELLIGENCE AGENCY
 INFORMATION FROM
 FOREIGN DOCUMENTS OR RADIO BROADCASTS

REPORT
 CD NO.

COUNTRY USSR

DATE OF
 INFORMATION 1950

SUBJECT Scientific - Metals, testing, plating,
 anodizing

HOW
 PUBLISHED Monthly periodical

DATE DIST. 17 Nov 1951

WHERE
 PUBLISHED Moscow

NO. OF PAGES 4

DATE
 PUBLISHED Dec 1950

LANGUAGE Russian

SUPPLEMENT TO
 RE. RT NO.

THIS DOCUMENT CONTAINS INFORMATION AFFECTING THE NATIONAL DEFENSE
 OF THE UNITED STATES WITHIN THE MEANING OF ESPIONAGE ACT 46
 U. S. C., 31 AND 32, AS AMENDED. ITS TRANSMISSION OR THE REVELATION
 OF ITS CONTENTS IN ANY MANNER TO AN UNAUTHORIZED PERSON IS PRO-
 HIBITED BY LAW. REPRODUCTION OF THIS FORM IS PROHIBITED.

THIS IS UNEVALUATED INFORMATION

SOURCE Zavodskaya Laboratoriya, No 12, 1950, pp 1433-1435

50X1-HUM

APPLICATION OF OPTICAL-MECHANICAL AND INTERFERENCE-CONTACT METHODS
 FOR MEASURING THE THICKNESS OF ELECTROPLATING AND OXIDE FILMS

I. A. Makolkin

[Figure referred to is appended.]

The optical method of measuring the thickness of the layer on a metal surface [1] permits the detection of a 5- to 10-A increase in surface film thickness. However, the upper limit for measurement by this method is limited (300 A). This factor prohibits utilization of the method for determining the thickness of a film obtained by anodization. It is also useless in other investigations which involve the measurement of film thickness in microns or tenths of a micron.

Optical-mechanical and interference-contact methods have been used for these purposes. The first of these methods involves the use of an ultraopti-meter; the second makes use of a microinterferometer of the Engineer Uverskiy type. Both devices have previously been described in detail [2, 3, 4].

The apparatus is designed for comparative contact measurements of plane-parallel end gages, and of various parts having planes which are finished plane-parallel to tenths or hundredths of a micron. This pertains then, to electroplating and to oxide layers formed at high temperatures.

It should be noted that it is difficult to achieve accurate finishing in the case of nonferrous alloys.

With regard to measuring the thickness of electroplated coatings on any sample (which we shall call the control sample), made not only of steel, but also of nonferrous metal (e.g., copper or brass), during the deposition of metal layers of the order of 10-15 μ , it is possible to limit the finishing of the control sample to plane-parallel down to $\pm 0.5\mu$. In this case, measurement error will amount to 3-5%. If the drop method is used, error will amount to 15-20%; and if any of the various magnetic-electric thickness gages is used, this error will be 20-30%.

- 1 -

CLASSIFICATION		DISTRIBUTION		CONFIDENTIAL			
STATE	<input checked="" type="checkbox"/> NAVY	<input checked="" type="checkbox"/> NSRB					
ARMY	<input checked="" type="checkbox"/> AIR	<input checked="" type="checkbox"/> FBI	are	<input checked="" type="checkbox"/>			

CONFIDENTIAL

50X1-HUM

In measuring a thickness expressed in tenths of a micron, it is best to select several sections on the surface of the sample and finish these sections plane-parallel to $0.1-0.2\mu$.

Experience indicates that control samples should measure $35 \times 5 \times 5 \text{ mm}^3$, which will assure the possibility of accurate finishing and prevent the sample from sagging, particularly if the sample is subjected to high-temperature heating.

The parts to be coated are submerged in the electroplating bath along with the steel control sample, which has been previously prepared in the same manner as an ordinary production part. It is necessary that the control sample be placed in a central part of the bath and not covered by another piece in the batch.

The absolute thickness of the control sample is measured at two or three points on an ultramicrotometer or Uverskiy interferometer before and after coating.

Half the difference between the beginning and end thicknesses determines the thickness of the metal coating, which thickness is equivalent practically to that of metal coating on production parts.

Measurements of coating thickness were made during the deposition on copper samples of a layer of copper at $t=20^\circ$, $D_k=2 \text{ a/sq dm}$; a layer of nickel at $t=40^\circ$, $D_k=2.5 \text{ a/sq dm}$; and a layer of chromium at $t=45^\circ$, $D_k=15 \text{ a/sq dm}$.

Steel third-class Johansson blocks were used as samples for nickel and chrome plating in control experiments. The surface of the blocks was finished plane-parallel to within hundredths of a micron.

The results are given in Table 1.

Table 1. Experimental Results

<u>Deposition of Metal on Sample</u>	<u>Thickness of Coating (μ)</u>	<u>Max Diff in Measurements of Coating Thickness (%)</u>	<u>Methods Compared</u>
Copper on copper	5-20	6	Gravimetric and optical-mechanical
Nickel on copper	3-11	7	Gravimetric and interference-contact
Chrome on copper	~1 4-12	20 5	Same Same
Nickel and chromium on Johansson block	2.5-8	13	Same

As shown in Table 1, the difference in measurements of coating thickness by the various methods lies within the limits of 13% for a coating of $2.5-20\mu$.

The measurement of the thickness of the oxide film during the anodization of aluminum and duralumin is of particular interest.

Three samples of aluminum and four samples of duralumin were subjected to anodization in a 20% solution of sulfuric acid at $D_A = 1-3 \text{ a/sq dm}$.

- 2 -

CONFIDENTIAL

CONFIDENTIAL

50X1-HUM

The thickness of the film was measured interferometrically and on the metal microscope.

Results of the measurements are shown in Table 2.

50X1-HUM

Table 2.

No	Anodi- zation (min)	Abs Thickness of Samples (mm)		Abs Thickness of Oxide Film (μ) by		
		Before Anodi- zation	After Anodi- zation	Inter- fero- meter	Metal Micro- scope	Diff (μ)
1	40	8.3760	8.3805	2.25	6.4	4.15
2	40	8.3112	8.3170	2.9	8.2	5.3
3	40	8.3150	8.3200	2.5	7.3	4.8
4	40	8.3154	8.3224	3.5	9.5	6.0
5	40	7.2860	7.3100	12.0	26.0	14.0
6	60	7.2270	7.2760	24.5	55.0	30.5
7	60	7.2780	7.3320	27.0	58.0	31.0

The data in Table 2 show that in the anodization of aluminum and duralumin, since the volume of the oxide is greater than the volume of the oxidized metal, the sample's absolute dimensions increase. The theory of the phenomenon of the growth of anodic oxide films has been worked out in experiments [57].

From the data in Table 2, a diagram of the growth of the oxide film on aluminum has been constructed, and is shown in Figure 2.

It can be seen from the diagram that:

$$\delta_2 = \delta - \frac{D_2 - D_1}{2},$$

where D_1 and D_2 are diameters of the sample before and after anodization; δ is the true thickness of the oxide film; δ_2 is the increase in the thickness of the aluminum.

Attention was also devoted to measuring the thickness of an oxide film produced during the heating of an alloy of magnesium with aluminum (9.44% Al; 0.59% Zn; 0.3% Mn). Heating was carried on for 16 hr at a temperature of 460°. Results of the measurements are shown in Table 3.

Table 3.

Absolute Thickness of Samples (mm)		Absolute Thickness of Oxide Film (μ), Measured by		
Before Expt	After Expt	Inter- fero- meter	Metal Micro- scope	Gravi- metric Method
11.5260	11.5360	5.0	10.9	9.09
11.5140	11.5230	4.5	10.0	9.16
11.3140	11.3236	3.8	10.6	9.08
11.3846	11.3938	5.6	11.2	9.04
11.4253	11.4347	4.7	9.7	9.02
11.4728	11.4826	4.9	10.3	9.05

CONFIDENTIAL

CONFIDENTIAL

50X1-HUM

It follows from Table 3 that measurements of the true thickness of the film made on the metal microscope with the aid of an eyepiece micrometer and calculated from gravimetric data obtained on an adsorption balance are in good agreement with each other.

Interferometer measurements of the thickness of the same films show values only half as great.

This is understandable since the optical-mechanical and interference-contact methods permit measurement of only that part of the thickness of the film which increases the external dimensions of the sample, but does not include the film below the initial level of the metal surface.

Conclusions

1. The applicability of optical-mechanical and interference-contact methods for measuring electroplated coatings on metals was shown using metal films of copper, nickel, and chromium.
2. The applicability of these methods to measuring the thickness of oxide films produced by anodization and thermal oxidation was shown by means of a study of oxide films on aluminum and magnesium alloys.

BIBLIOGRAPHY

1. Krylova, Izv. AN SSSR, OTN, No 10, 1938.
2. I. A. Grigor'yev, Technical Measurements in Machine Building, p 100 (1948).
3. Symposium of Materials and Instructions, Control of Means for Measuring Dimensions in Machine Building, p 394, (1948)
4. Instruction for Using an Interferometer With a Variable Scale on the Engineer I. T. Uverskiy System, "Kalibr" Plant, 1949.
5. G. V. Akimov, N. D. Tomashov, M. N. Tyukina, Zhurnal Obshchey Khimii, No 9, 10, 11, 12, 1942.

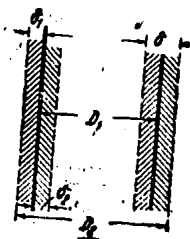


Figure 2. Diagram of Oxide Film Growth on Aluminum During Anodization

- E N D -

- 4 -

CONFIDENTIAL